Unloading Effect on Delayed Hydride Cracking in Zirconium Alloys

Young Suk Kim [a*], Sung Soo Kim [a]
[a] *Hydrogen Cracking Team, Korea Atomic Energy Reseach Institute, 150, Dukjin-dong, Yuseong, Daejeon 305-353, Republic of Korea*
[*] *Corresponding author: yskim1@kaeri.re.kr*

1. Introduction

It is well-known that a tensile overload retards not only the crack growth rate (CGR) in zirconium alloys during the delayed hydride cracking (DHC) tests but also the fatigue crack growth rate in metals, the cause of which is unclear to date. A considerable decrease in the fatigue crack growth rate due to overload is suggested to occur due either to the crack closure [1] or to compressive stresses or strains [2] arising from unloading of the overload. However, the role of the crack closure or the compressive stress in the crack growth rate remains yet to be understood because of incomplete understanding of crack growth kinetics. The aim of this study is to resolve the effect of unloading on the CGR of zirconium alloys, which comes in last among the unresolved issues as listed above. To this end, the CGRs of the Zr-2.5Nb tubes were determined at a constant temperature under the cyclic load with the load ratio, R changing from 0.13 to 0.66 where the extent of unloading became higher at the lower R. More direct evidence for the effect of unloading after an overload is provided using Simpson's experiment [3] investigating the effect on the CGR of a Zr-2.5Nb tube of the stress states of the pre-fatigue crack tip by unloading or annealing after the formation of a pre-fatigue crack.

2. Experimental Procedures

The DHC tests were conducted at a constant temperature of 250°C under the cyclic and constant loads on the 17 mm compact tension (CT) specimens taken from a heat-treated Zr-2.5Nb tube [4]. These specimens were pre-charged to 60 ppm of hydrogen using an electrolytic method followed by homogenization treatments and then pre-fatigued to introduce a 1.7mm fatigue crack. A thermal cycle was applied during the DHC tests where the test temperature of 250°C was approached by cooling from 315°C at a cooling rate of 1.5°C/min. Then, the tension-tension cyclic loads were applied with 1 cycle/min to the CT specimens where load ratio, R was varied from 0.13 to 0.68 by changing the minimum load. Furthermore, the constant load termed R=1 was applied to investigate the effect of the absence of unloading on the CGR. The detailed test conditions for cyclic loading are given in [4]. Crack growth during the DHC tests was monitored using a potential drop versus crack length calibration curve. After completion of the DHC tests, the actual crack lengths were determined from the fracture surfaces using a stereoscope.

To corroborate the effect of unloading, the CGR of a cold-worked Zr-2.5Nb tube (CANDU type) has been determined under the constant load at 120°C with and without unloading from 15 MPa√m, or after annealing at 475°C [3]. The DHC tests were conducted on the as-received compact tension (CT) specimens taken from the CANDU Zr-2.5Nb tube with around 10 ppm H with the test temperature being approached by heating. The applied stress intensity factor K_I starting from <6 MPa√m increased in steps of 1 to 1.5 MPa√m until cracking commenced. To change the stress state at a crack tip, three different procedures were applied: the first one was to produce a pre-fatigue crack under a maximum final K of <5 MPa√m, the second was to produce a pre-fatigue crack under a maximum final K of <5 MPa√m followed by annealing at 475°C to relieve any residual stresses remaining at the pre-fatigue crack tip, and the third was to apply unloading when K_I had reached about 15 MPa√m and then to reapply the load in steps [9].

3. Results and Discussion

The CGR at 250°C of the heat-treated Zr-2.5Nb tube was the highest under the constant load and decreased under the cyclic load with decreasing R, as shown in Fig. 1. Given the absence of unloading under the constant load, it is evident that the highest CGR under the constant load is due to the absence of unloading during the DHC tests. In contrast, the extent of unloading that increased with decreasing R under the cyclic load was the cause of a linear dependence of the CGR on the R, as shown in Fig. 2. To assess the effect of the cyclic load on the cracking of hydrides precipitated at a crack tip, the striation spacing was measured as a function of R. As shown in Figs. 3, the striation spacing became narrower with decreasing R. Given that the striation spacing represents the critical hydride length above which the cracking of hydrides occurs [5,6], it is obvious that the critical hydride length became smaller with decreasing R. In other words, the cyclic load enhances the rate of hydride cracking when compared to the constant load, causing the hydrides to easily fracture at a smaller length. The results of Figs. 2 to 3 showed that the cyclic load enhanced the rate of hydride cracking but decreased the CGR. In other words, in the cyclic load, the CGR decreased with decreasing R despite the enhanced rate of hydride cracking. This fact demonstrates that it is not the rate of hydride cracking but the rate of hydride nucleation that governs the CGR according to Kim's model [7-10]

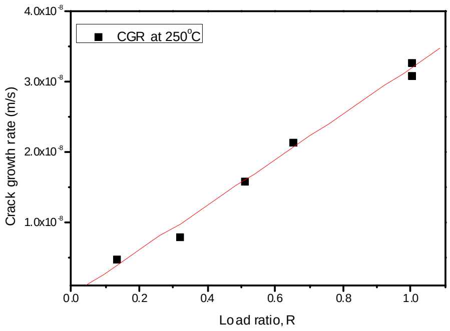

Fig.1. DHC crack growth rate of a heat-treated Zr-2.5Nb tube at 250°C under the constant and cyclic loads with the load ratio, R changing from 0.13 to 0.68.

given that the rate of hydride growth that is constant at a constant temperature does not change independent of R. As the extent of unloading arising from the cyclic load increases with decreasing R, a linear dependence of the CGR on the R as shown in Fig. 1 corroborates that the CGR is retarded in proportion with the extent of unloading. Hence, considering that nucleation of hydrides is restrained by the compressive stress, unloading in the cyclic load induces the compressive stress at a crack tip [2] the magnitude of which increases with decreasing R, suppressing the rate of hydride nucleation so as to cause it to become the slowest among the three processes involved in DHC. This rationale explains a linear dependence of the CGR on the R (Fig. 1) despite the faster rate of hydride cracking with decreasing R, as shown in Fig. 1, which arises due to an increase in the compressive stress with the R.

Definitive evidence for the effect of unloading is provided by Simpson's experiment [3] where the CGRs of a cold-worked Zr-2.5Nb (termed CANDU) tube were determined under the constant load at 120°C without and with unloading from 15 MPa√m and with annealing at 475°C after the formation of a pre-fatigue crack. Without loading, as shown in Fig. 2, the CGR of the CANDU Zr-2.5Nb tube was 2.1×10^{-9} m/s independent of K_I above K_I of 10 MPa√m. In contrast, after unloading from 15 MPa√m, for example, it decreased to 8.4×10^{-10} m/s at 14.9 MPa√m (corresponding to point A in Fig. 6), then increased rapidly with increasing K_I and became constant at K_I of over 18 MPa√m (Fig. 2). However, annealing decreased the K_{IH} to around 6 MPa√m and increased the CGR to, for example, 3.1×10^{-9} m/s at 14.9 MPa√m, as indicated by the red vertical line in Fig. 6. The results of Fig. 4 clearly showed that unloading decreased the CGR and increased K_{IH} when compared to those without unloading. Therefore, it is definitively evident that a decrease of the CGR under the cyclic load as shown in Fig. 1 is due to unloading.

4. Conclusions

The CGR at 250°C of a heat-treated Zr-2.5Nb tube was the highest under the constant load and decreased under the cyclic load with decreasing load ratio, R due to unloading. Given the presence of unloading during

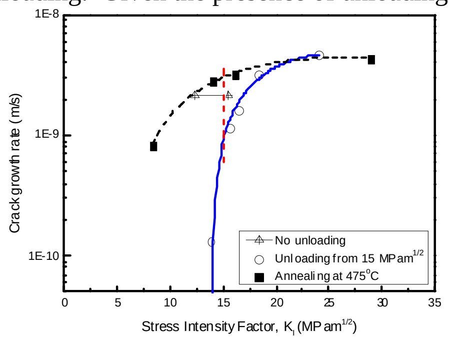

Fig. 2. Crack growth rate of a cold-worked Zr-2.5Nb tube without and with unloading from 15 MPa√m, and with annealing at 475°C after pre-fatigue cracking (reproduced from [3]).

the cyclic load, the decreased CGR under the cyclic load is due to unloading in the cyclic load inducing the compressive stress at the crack tip. This compressive stress suppresses the hydride nucleation rate, leading it to govern the CGR, according to Kim's new model. Definitive evidence is provided using Simpson's experiment. Consequently, it is demonstrated that the compressive stress induced by unloading suppresses the hydride nucleation rate and hence, the CGR under the cyclic load and annealing does the reverse.

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